Measurement under pressure of thermoelectric power along the thickness of a thin specimen

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A method of measuring under pressure the thermoelectric power along the thickness of thin specimens is described. This method is useful in measuring thermoelectric power in a direction perpendicular to the cleavage plane of a crystal.

The high pressure cell for tungsten carbide opposed anvil setup described earlier[1] has been used successfully to measure thermoelectric power (TEP) across the phase transitions of a number of solids.[2–4] The method requires samples approximately 4 mm long, 1 mm wide and 0.1 mm thick and the TEP measured is along the length of the specimen. In this note a method is described to measure TEP along the thickness of the specimen. The method is useful in measuring TEP in a direction perpendicular to the cleavage plane of a crystal.

A pair of tungsten carbide anvils (without any binding rings, 25.4 mm diameter, 40 mm length, flat face diameter 12.7 mm) powered by a 100 ton hydraulic ram is used to generate pressure. The gasket assembly is shown in Fig. 1. A pyrophyllite gasket (12.5 mm o.d., 5 mm i.d., 0.35 mm thick) with a closely fitting talc disk at the center is placed on the anvil. A 0.1-mm-deep diametric groove is made on the top surface of the gasket. A chromel-alumel thermocouple (0.1 mm diameter), A_1–C_1 is placed in the groove such that the thermojunction, J_1, is at the center of the talc disk. A specimen measuring approximately $2\times2\times0.4$ mm^3 is placed at the center of another pyrophyllite gasket (12.5 mm o.d., 5 mm i.d., 0.4 mm thick), and the annular space around the specimen filled with 1:1 volume mixture of epoxy and talc powder. After allowing sufficient time (12–24 h) for the epoxy to set, the gasket-specimen assembly is thinned to 0.25 mm by working on both sides on 600 grade emergy paper, such that the specimen surface is exposed on both sides. The gasket-specimen assembly is placed atop the first. The thermojunction, J_1, comes in contact with the bottom surface of the specimen. A third gasket with a talc disk at the center, dimensions being the same as that of the first gasket, was placed atop the second gasket. A thermocouple, A_2C_2, was placed between the second and third gasket, with the thermojunction, J_2, in contact with the top surface of the specimen. A temperature gradient along the thickness of the specimen is set up with the help of a 5 W heater wound around the lower anvil. The temperature difference up to 2 K can be set up between the two faces of the sample. A larger temperature difference is difficult to achieve because of good thermal contact (through the gasket) between the anvils. The specimen temperature can be varied between room temperature and 100 °C by placing a tubular heater (not shown in Fig. 1) around both the anvils. At a higher temperature the epoxy in the epoxy-talc mixture around the specimen softens and often results in a blowout of the gasket. Temperatures below room temperature, down to 77 K, have been achieved by immersing the anvils in liquid nitrogen. The voltages ΔE_{AA} and ΔE_{CC}, respectively, between two alumel (A_1–A_2) and two chromel (C_1–C_2) leads are measured using a Keithley-196 digital multimeter. For higher precision a nanovolt potentiometer (7555-K-5, Leeds & Northrup) can be used. The TEP of the specimen, Q_s, is calculated using the following relation:

$$Q_S = (Q_A - rQ_C)/(1 - r),$$

where Q_A and Q_C are, respectively, the absolute TEP of alumel and chromel, and $r = \Delta E_{AA}/\Delta E_{CC}$. Though we have used dc-voltage measurement to obtain TEP, in principle more sensitive techniques that use computers can be interfaced with this cell.[5–8] However, ac-voltage measurements cannot be used here due to the large mass of the anvils.

The load applied on the anvil was calibrated in terms of specimen pressure by the procedure discussed elsewhere. The highest pressure reached with this setup was 6 GPa. The performance of the high pressure cell was assessed by measuring the TEP of mercury selenide as a function of pressure. Since the zinc-blende-to-cinnabar transition[9,10] is sluggish the experiments were done at 90 °C by heating the anvils with a tubular heater. Even at this temperature, transition took 5 h for completion. The variation with pressure of TEP indicated by the present data (Fig. 2) agrees well with that

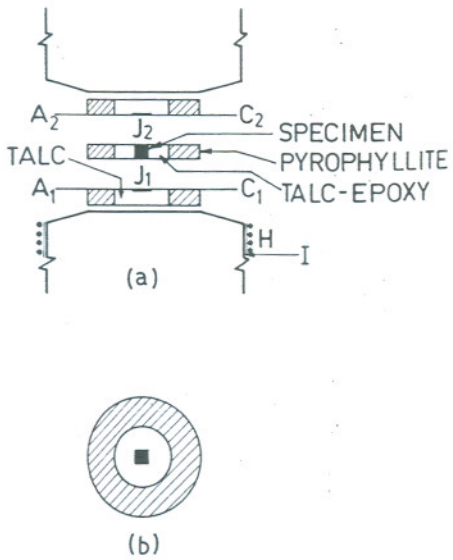

FIG. 1. (a) Schematic diagram of the high pressure cell. J_1, J_2—thermocouple junctions. A_1C_1, A_2C_2—thermocouples. H—heater, I—insulation. (b) Pyrophyllite gasket containing the sample set in talc epoxy.

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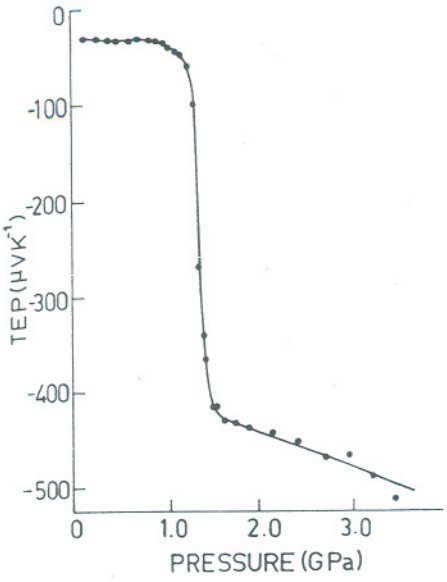

FIG. 2. TEP vs pressure of HgSe at 90 °C.

observed in an independent measurement with piston-cylinder apparatus.[11]

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